



Designation: D8363 – 20

Standard Test Method for Rubber—Stress Relaxation in Tension Under Non-Isothermal Conditions¹

This standard is issued under the fixed designation D8363; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method is used to determine the non-isothermal stress relaxation, also known as temperature scanning stress relaxation (TSSR). Stress relaxation is a characteristic behavior of rubber materials.

1.2 *Units*—The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

D3183 Practice for Rubber—Preparation of Pieces for Test Purposes from Products

D412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

¹ This test method is under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and is the direct responsibility of Subcommittee D11.10 on Physical Testing.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3. Terminology

3.1 Definitions:

3.1.1 *standard laboratory temperature, n*—temperature of $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$).

3.1.2 *stress relaxation, n*—decrease in stress after a given time at constant strain.

4. Summary of Test Method

4.1 This test method provides a procedure for non-isothermal stress relaxation as a function of temperature at constant elongation and at a specific heating rate.

4.2 This test method is useful for the accelerated determination of the thermal mechanical behavior of a rubber, for example, its ageing behavior or the parameters describing the limits for a thermal application of the material.

5. Significance and Use

5.1 Non-isothermal stress relaxation, also known as temperature scanning stress relaxation, performed at a specific heating rate, delivers a set of parameters useful to specify the properties of thermo-plastic elastomers. It can also characterize the deterioration of the cross-linked rubber network in a reasonable testing time of a few hours.

5.2 Stress relaxation tests are typically performed as time-dependent experiments at constant strain and temperature. It is known that temperature has a strong influence on the relaxation time of rubber. When evaluating ageing behavior such as deterioration of the network, a reliable test using isothermal stress relaxation requires extremely long testing times, for example, days or weeks depending on the application.

6. Apparatus

6.1 Tensile tester equipped with an oven according to Test Methods **D412** consisting of an electrically driven linear extension actuator equipped with two grips and capable of holding the test piece without slipping at a fixed extended length within $\pm 1\%$ accuracy. It shall be able to measure and record the force acting on the test piece with an accuracy of $\pm 1\%$.

6.2 The grips shall be positioned inside the oven for testing of a single test piece. The oven shall maintain a stable heating

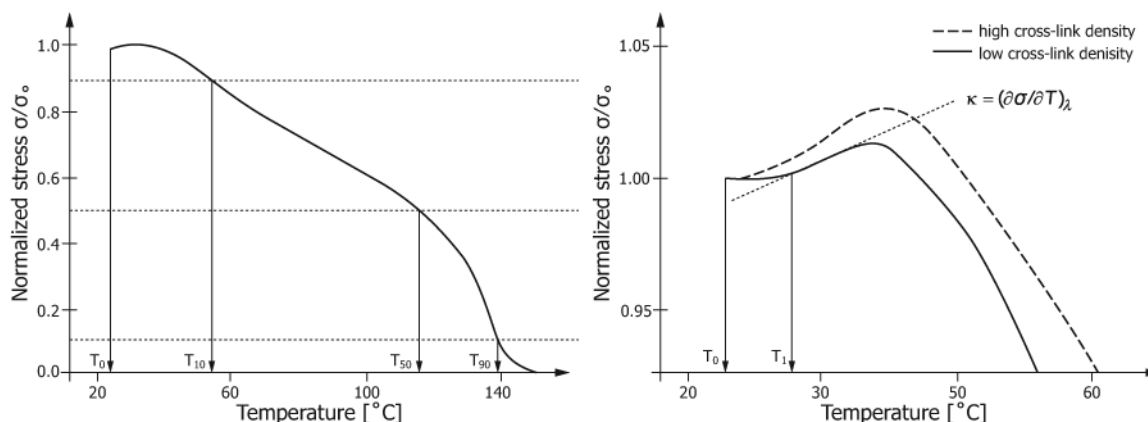


FIG. 1 Left (a) schema of a non-isothermal stress relaxation curve indicating the characteristic temperatures at a specific stress decay, and right (b) detail schema of the initial slope of the non-isothermal stress relaxation curve

rate of at least 2 ± 0.05 K/min ($^{\circ}\text{C}/\text{min}$) at the narrow position of the dumbbell during the test. The oven is equipped with a temperature sensor placed close to the surface of the test specimen.

7. Test Specimen

7.1 The test specimens shall be dumbbells with a standard thickness of 2.0 ± 0.2 mm and a length of 75 ± 0.5 mm. The dimension of the narrow portion of dumbbells shall be 2.0 ± 0.2 mm by 25 ± 0.5 mm by 4 ± 0.5 mm. Test specimens shall be cut from molded sheets prepared in accordance with Practice D3182 or from product pieces prepared according to Practice D3183.

7.2 Test specimens shall be cut with the die cutter parallel to the milling-grain direction. The die cutter shall be sharp and free from nicks and oil prior to cutting. Support the sheets on a suitable cutting surface (cardboard, linoleum, etc.) covered with a thin plastic film to prevent inclusions. Cut the sheets or pieces with a single, continuous stroke.

7.3 Discard test specimens having obvious flaws. Before testing, physically randomize the test specimens from all sheets of the same compound.

7.4 Test specimens shall be conditioned in an unstrained state for at least 24 h at standard laboratory temperature before testing. A minimum of three test specimens per compound is recommended for testing.

8. Procedure

8.1 Isothermal Conditioning:

8.1.1 Place the test piece between the grips of the tensile tester in the unstrained condition at the standard laboratory temperature.

8.1.2 Close the oven door and wait for a stable temperature reading at the narrow position of the dumbbell.

8.1.3 Extend the dumbbell to a recommended elongation of 50 % strain, ϵ_0 , within 1 min. If the test piece breaks, reduce the elongation stepwise by 10 % strain ϵ_0 until the test piece is able to withstand the applied strain or apply another appropriate elongation.

8.1.4 Record the isothermal force decay $F(t)$ at 23°C as a function of the time t for 2 h, preferably, or for another specified duration of the isothermal conditioning. This isothermal conditioning might have a strong influence on the results of the non-isothermal relaxation due to short-term relaxation processes. Final test results are only comparable if the tests are performed with identical duration of the conditioning isothermal relaxation.

8.2 Non-Isothermal Relaxation:

8.2.1 Record the non-isothermal force decay $F(T)$ as a function of the temperature, T . The initial value F_0 is the force at 23°C after completing the isothermal conditioning in 8.1.4.

8.2.2 Increase the temperature of the oven with a constant heating rate of 2 ± 0.05 K/min ($^{\circ}\text{C}/\text{min}$) or other required heating rates depending on the control stability of the oven. Results obtained with different heating rates are not comparable.

8.2.3 The sampling rate of the force data shall be at least 1 Hz for the heating rate of 2 K/min ($^{\circ}\text{C}/\text{min}$). If required, the sampling rate might be adapted to other heating rates.

8.2.4 Stop the test when the test piece breaks, the force reading is null, the force ratio $F(T)/F_0$ reaches a predetermined value (for example, 0.5 or 0.1), or when the designated final temperature has been reached.

9. Calculation or Interpretation of Results

9.1 *Parameters obtained from the non-isothermal stress relaxation curve*—The tensile stress $\sigma(T)$ is the force $F(T)$ normalized by the cross section A_0 of the test piece at the unloaded initial state. The initial tensile stress σ_0 is the stress measured at the corresponding initial temperature T_0 . The relaxation modulus $E(T)$ at a specified temperature T is given by the ratio of the stress $\sigma(T)$ and the nominal strain ϵ_0 .^{3,4}

³ Vennemann, N., Characterization of Thermoplastic Elastomers by Means of Temperature Scanning Stress Relaxation Measurements, in: A. Z. El-Sonbati (Ed.), *Thermoplastic Elastomers*, INTECH, Rijeka (Croatia), 2012, pp. 347-370.

⁴ Vennemann, N., Hündorf, J., and Kummerlöhne, C., Phase Morphology and Relaxation Behaviour of SEBS/PP Blends, *KGK Kautschuk Gummi Kunststoffe* 54, Jahrgang, Nr. 7-8, 2001, pp. 362-367.